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1. Your reference: AA 1692 GB

2. Patent application number *(The Patent Office will fill in this part)*: 0405015.9 - 5 MAR 2004

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*:
JOHNSON MATTHEY PUBLIC LIMITED COMPANY,
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Patents ADP number *(if you know it)*: 53626801 0.

If the applicant is a corporate body, give the country/state of its incorporation:

4. Title of the invention:
METHOD OF LOADING A MONOLITH WITH CATALYST AND/OR WASHCOAT

5. Name of your agent *(if you have one)*:
ANDREW DOMINIC NUNN

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*:
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Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

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Number of earlier application	Date of filing *(day / month / year)*

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Description	3
Claim(s)	1
Abstract	1
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Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	
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11. I/We request the grant of a patent on the basis of this application.

Signature [signature] Date 5/3/04

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AA 1692

METHOD OF LOADING A MONOLITH WITH CATALYST AND/OR WASHCOAT

The present invention relates to a method of loading a monolith with a catalyst and/or a washcoat and in particular to a method of inserting the catalyst and/or washcoat in the pore structure of the monolith.

It is known to load a catalyst and/or a washcoat on a honeycomb monolith such as a ceramic flow-through monolith or wall-flow filter. The resulting piece is then dried and calcined to make the desired product. See for example our PCT application claiming priority from GB 0304939.2.

A washcoat is generally a slurry comprising a high surface area particulate refractory oxide such as bulk ceria, silica, alumina, titania, zirconia, or a mixed oxide or composite oxide of any two or more thereof, e.g. ceria-zirconia, silica-alumina etc. The washcoat and/or the refractory oxide particles can include an active catalytic metal compound such as a platinum group metal, e.g. platinum, palladium or rhodium, or a molten salt to promote soot combustion e.g. an alkali metal salt or a lanthanum salt of vanadium, tungsten or molybdenum or vanadium pentoxide.

By "composite oxide" herein, we mean a largely amorphous oxide material comprising oxides of at least two elements which are not true mixed oxides consisting of at least two metals.

Alternatively, the monolith material itself can be impregnated with a suitable aqueous salt of any of the above metals and the resulting piece is then dried and calcined. Of course, a washcoated monolith that has been dried can also be impregnated using this method.

A typical wall-flow filter has a shape of a honeycomb, the honeycomb having an inlet end and an outlet end, and a plurality of cells extending from the inlet end to the outlet end, the cells having porous walls wherein part of the total number of cells at the inlet end are plugged along a portion of their lengths, and the remaining part of the cells

that are open at the inlet end are plugged at the outlet end along a portion of their lengths, so that a flowing exhaust gas stream passing through the cells of the honeycomb from the inlet end flows into the open cells, through the cell walls, and out of the filter through the open cells at the outlet end.

Ceramic materials for flow-through monoliths and wall-flow filters are typically made of at least one of silicon, silicon carbide, aluminium nitride, silicon nitride, aluminium titanate, alumina, cordierite, mullite pollucite or a thermet such as Al_2O_3/Fe, Al_2O_3/Ni or B_4C/Fe.

It is also known that catalysed soot filters require more porosity and generally larger pore sizes to non-catalysed filters to enable coating with catalyst systems. In order to have acceptably low pressure losses after being coated with the catalyst/washcoat systems at about 50 g/dm^3 loading. Typical porosity is usually from about 45-55%. Where the catalyst system comprises a NO_x storage/reduction system, higher washcoat loadings are required, possibly above 100 g/dm^3. In this embodiment, filter substrate porosity may be above 60%.

One method of loading the pore structure of a wall-flow filter is disclosed in EP 0766993. One end of a honeycomb monolith is alternately plugged as described above. The plugged end is labelled the exhaust gas outlet end and is disposed with the plugged end uppermost. A washcoat composition is applied to plugged end which flows down the channels and permeates into the porous walls due to capilliarity. To facilitate this process, the coating solution may be sucked through the monolith under vacuum. The resulting piece is dried, the other end of the monolith is plugged to generate a wall-flow filter having the above described structure.

We have considered this method and do not believe it is of practical utility for a number of reasons. Firstly, the method is very labour intensive requiring a number of separate steps in order to generate the desired piece. For example, a better method would load a catalyst and/or washcoat on an unloaded wall-flow filter, i.e. wherein both ends are already plugged. Secondly, the use of a vacuum does not guarantee insertion of the desired washcoat components in the pore structure of the filter. In particular, we have found that

by applying a vacuum washcoat components can build up in a cake, proventing satisfactory ingress of the desired components into the pore structure of the monolith. However, relying on capilliarity to introduce washcoat components in the pore structure , particularly for more viscous washcoats, is time intensive.

We have now developed a method of loading a monolith with a catalyst and/or a washcoat wherein the problems associated with this prior art are reduced or avoided. In one embodiment the method is of particular utility to wall-flow filters, although the method can also be used with advantage for loading through-flow monolith substrates.

According to one aspect, the invention provides a method of loading a monolith with a catalyst and/or a washcoat, which method comprising introducing the monolith into a closeable enclosure, applying a vacuum to the enclosure to withdraw gas from a pore structure of the monolith and introducing a liquid comprising the catalyst and/or washcoat into the vacuated enclosure to insert the catalyst and/or washcoat in the monolith pore structure.

An advantage of the present invention is that, by removing the air from the pore structure of the monolith, we have found that the ingress of liquid is greatly facilitated.

In a further aspect the invention provides an apparatus for carrying out the method according to the invention, comprising a closeable enclosure and means for introducing a liquid into the vacuated enclosure.

CLAIMS:

1. A method of loading a monolith with a catalyst and/or a washcoat, which method comprising introducing the monolith into a closeable enclosure, applying a vacuum to the enclosure to withdraw gas from a pore structure of the monolith and introducing a liquid comprising the catalyst and/or washcoat into the vacuated enclosure to insert the catalyst and/or washcoat in the monolith pore structure.

2. Apparatus for carrying out the method according to claim 1, comprising a closeable enclosure and means for introducing a liquid into the vacuated enclosure.

ABSTRACT

METHOD OF LOADING A MONOLITH WITH CATALYST AND/OR WASHCOAT

A method of loading a monolith with a catalyst and/or a washcoat comprises introducing the monolith into a closeable enclosure, applying a vacuum to the enclosure to withdraw gas from a pore structure of the monolith and introducing a liquid comprising the catalyst and/or washcoat into the vacuated enclosure to insert the catalyst and/or washcoat in the monolith pore structure. An apparatus for carrying out the method comprises a closeable enclosure and means for introducing a liquid into the vacuated enclosure.